GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[X] ORIGINAL

[ ] REVISION NO. ____

Project No. A-2911 DATE: 5/25/81

Project Director: Mr. J. N. Harris School/Lab EMSL/MRB

Sponsor: Selenia SpA; Rome, Italy

Type Agreement: Standard Industrial dated 3/12/81, with P. O. No. 018104977

Award Period: From 5/1/81 To 7/31/81* (Performance) ----- (Reports)

Sponsor Amount: $10,989 Contracted through:

Cost Sharing: None GTRI/~~XXX~~

Title: Characterization of Properties of High Purity Fused Silica Slips

ADMINISTRATIVE DATA OCA CONTACT Duane Hutchison x 4820

1) Sponsor Technical Contact: Mr. R. Brugiotti, Technical Manager; Selenia SpA; Via Tiburtina; Km. 12.400; 00131 Rome, Italy.

2) Sponsor Admin./Contractual Contact:

Reports: See Deliverable Schedule Security Classification: None

Defense Priority Rating: None

RESTRICTIONS

See Attached N/A Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval - Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with N/A; none authorized/proposed

COMMENTS: * Project Director anticipates completion of work by 6/30/81

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GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: 6/24/81

Project Title: Characterization of Properties of High Purity Fused Silica Slips

Project No: A-2911

Project Director: Mr. J. N. Harris

Sponsor: Selenia Spa; Rome, Italy

Effective Termination Date: 7/31/81

Clearance of Accounting Charges: 7/31/81

Grant/Contract Closeout Actions Remaining:

X Final Invoice ~~and Closing Documents~~ thru GTRI (Letter of Credit Arrangement)

_ Final Fiscal Report

_ Final Report of Inventions

_ Govt. Property Inventory & Related Certificate

_ Classified Material Certificate

_ Other _____

Assigned to: EMSL/MRB (~~School~~/Laboratory)

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Georgia Institute of Technology
ENGINEERING EXPERIMENT STATION
ATLANTA, GEORGIA 30332

June 16, 1981

Selenia Industrie Electtroniche Associate SpA
Via Tiburtina
Km. 12.400
00131 Rome, Italy

Attention: Mr. R. Brugiotti, Technical Manager

Subject: Technical Letter Report: Characterization of High Purity Fused Silica Slip 042781-HPA

Gentlemen:

The rheological characteristics of the high purity fused silica slip 042781-HPA from Thermo Materials Corporation, Atlanta, Georgia, were evaluated to determine the acceptability of the slip per specifications of Selenia Industrie Elettroniche Associate SpA.

The characterization procedure shown in Figure 1 was followed to determine the rheological properties, the casting of test bars, and the sintering conditions. The high purity slip was packaged in a five gallon polyethylene carboy with a diameter of 38.9 centimeters. The speed of the roll mill was adjusted to produce a surface velocity of carboy of one meter per second. After the initial rolling time period of forty-eight hours, measurements were made for solids content, viscosity and pH. Rolling of the carboy was continued for cumulative times of 72 and 96 hours with pH and viscosity measurements made at each interval. After 96 hours, when pH and viscosity were constant values, eighty (80) 19.1-millimeter diameter test bars were cast.

The test bars were dried for twenty-four hours at 150^{o} C. Test bar sets of ten (10) each were sintered of temperatures of 1200^{o} and 1215^{o} C for four hours. The sintered bars were used to determine dynamic elastic modulus, modulus of rupture, bulk density, porosity, and cristobalite content. The slip test procedures are as follows:

I. Solids Content. The solids content was measured by evaporating the liquid from approximated 50-grams of slip to a constant weight in a 150^{o} C oven.

II. pH. The pH was measured by immersing the glass electrodes of the pH meter directly in the slip which had been temperature stabilized to 20^{o} C.

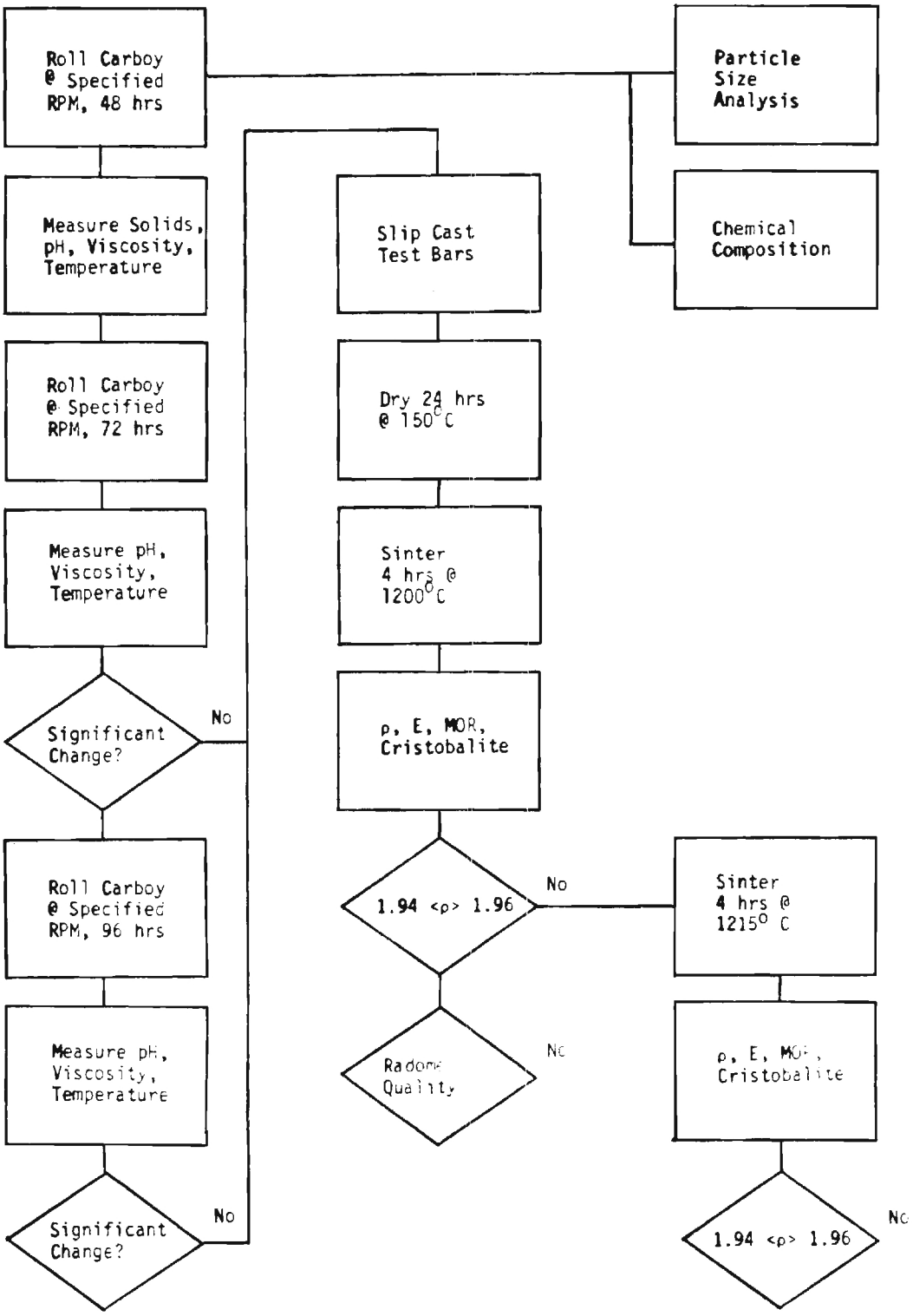

Figure 1. Flow Chart for the Characterization of a High Purity, Fused Silica Slip for Radome Applications.

III. Viscosity. The apparent viscosity was measured with a model LVF Brookfield Viscometer with a number 2 spindle. Measurements were made at speeds of 6, 12, 30 and 60 rpm. The temperature of the slip was stabilized at 20° C from a carboy rolling temperature above 25° C.

IV. Particle Size Distribution. The particle size distribution was measured by Micromeritics Instrument Corporation (MIC) Atlanta, Georgia, using the MIC Sedigraph 5000 and was supplied by the vendor. Data are provided as a plot of cumulative mass percent versus equivalent spherical diameter.

V. Chemical Composition. These data were determined from Spectro Chemical Laboratories, Pittsburgh, Pennsylvania, and were provided by the vendor.

VI. Bulk Density and Porosity. These properties were determined in accordance with ASTM C373 "Standard Method of Test for Water Absorption, Bulk Density, Apparent Porosity and Apparent Specific Gravity."

VII. Elastic Modulus. This property was determined in accordance with ASTM C623 "Standard Method of Test for Young's Modulus, Shear Modulus, and Poisson's Ratio for Glass and Glass Ceramics by Resonance."

VIII. Modulus of Rupture. This property was determined by a procedure modification for ASTM C158 "Flexure Testing of Glass." The test bars were broken in four point, 1/4-point configuration with a 102 millimeter lower span and a 51-millimeter upper span. The loading rate of the hydraulic Universal Test Machine was 600-pounds (272.4 kilograms) per minute.

IX. Cristobalite Content. The percentage of α cristobalite contained in the sintered specimens was determined by an x-ray analysis technique developed by the Georgia Institute of Technology. The results are based on a comparison with an industry accepted standard, Georgia Tech A-4.

The solids content for the slip was found to be 82.70 percent after a rolling time of 48 hours; this rolling time was selected based on results obtained from fused silica studies conducted for Selenia. The results of the rolling time versus slip stabilization are shown in Table I. The chemical composition of the high purity slip is shown in Table II and the particle size distribution is shown in Figure 2.

TABLE I

SLIP PROPERTIES FOR THERMO MATERIALS
HIGH PURITY FUSED SILICA SLIP 042781-HPA

Solid Content (%)	82.70
pH @ 20° C	
after 48 hrs	5.00
after 72 hrs	4.75
after 96 hrs	4.77
Viscosity @ 20° C (centipoise)	
after 48 hrs @ 6 rpm	125
12 rpm	130
30 rpm	153
60 rpm	137
after 72 hrs @ 6 rpm	95
12 rpm	138
30 rpm	145
60 rpm	139
after 96 hrs @ 6 rpm	100
12 rpm	143
30 rpm	144
60 rpm	138
Mean Particle Size (μm)	7.8

TABLE II

CHEMICAL COMPOSITION OF THERMO MATERIALS
HIGH PURITY FUSED SILICA SLIP 042781-HPA

	Weight Percent
Al_2O_3	0.25
TiO_2	0.002
Fe_2O_3	0.008
MgO	0.008
CaO	0.007
CoO	0.001*
Cr_2O_3	0.001*
SiO_2	99.72
	Parts Per Million
Na	10*
K	10*
Li	10*

*Not detected. The number indicates the minimum limit of detection.

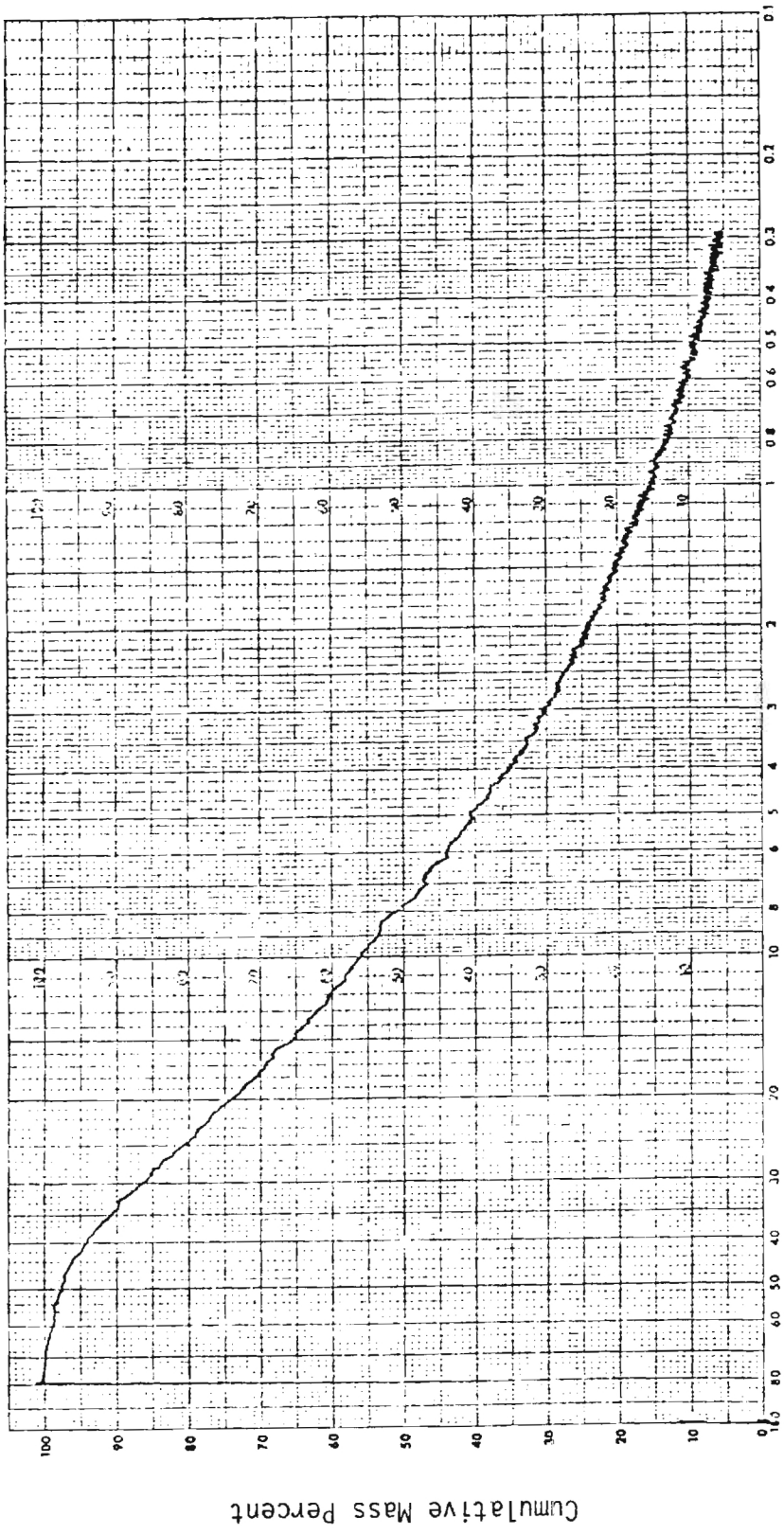

Figure 2. Particle Size Distribution of High Purity Fused Silica Slip 042781-HPA.

The sintering operations were performed in a bottom-loading electric-resistance furnace with the bars in a vertical position supported in a fused silica foam block. The sintering schedule was programmed by microprocessor control and is shown in Table III.

TABLE III

SINTERING SCHEDULE FOR HIGH PURITY
SLIP-CAST FUSED SILICA TEST BARS

Room Temperature to 927° C*	10 hours
Hold at 925° C*	6 hours
927° C to 1038° C	90 minutes
Hold at 1038° C	2 hours
1038° C to Sintering Temperature**	90 minutes
Hold at Sintering Temperature	4 hours
Turn off Kiln and Cool Bars in Kiln	

*Typical, can vary.

**Sintering #1 - 1200° C
Sintering #2 - 1215° C

The test results for the physical and mechanical properties obtained from the two sintering temperatures, 1200° and 1215° C, are shown in Table IV.

X-ray diffraction analyses for bulk α-cristobalite content of the mean, high, and low strength test bars in the 1200° C sinter temperature indicated values of 0.2 to 0.3 percent. This low amount of reported α-cristobalite is not usually reported with confidence since the amorphous background in x-ray analysis makes detection of levels below 0.5 percent uncertain.

The results of the rolling study indicate that the fused silica slip should be rolled at a surface velocity of one meter per second for a minimum of 72 hours to obtain slip stability. A sintering temperature of 1200° C should

TABLE IV

PHYSICAL AND MECHANICAL PROPERTIES OF
TEST SPECIMENS SINTERED 4 HOURS

	Sintering Temperature	
	1200^{o} C	1215^{o} C
Elastic Modulus (10^6 psi)	5.01 $\pm$ 0.14	5.78 $\pm$ 0.18
Modulus of Rupture (10^3 psi)	5.18 $\pm$ 0.35	6.66 $\pm$ 0.86
Bulk Density (g/cm^3)	1.937 $\pm$ 0.004	1.958 $\pm$ 0.003
Porosity (%)	11.35 $\pm$ 0.19	10.64 $\pm$ 0.15

result in physical and mechanical properties within the specifications of Selenia. This slip is satisfactory for radome manufacture.

Respectfully submitted,

Joe N. Harris
Project Director
Senior Research Engineer

A. T. Sales
Research Engineer II

Reference: Georgia Tech Research Notebook A-2347 - A-2911

jw